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IS 8195 (2004): 4-nitro Chlorobenzene [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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IS : 8195 - 1976

Indian Standard
**SPECIFICATION FOR
4-NITRO CHLOROBENZENE**

UDC 667.285.1 : 547.548



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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

November 1976

**AMENDMENT NO. 1 MARCH 2002
TO
IS 8195 : 1976 SPECIFICATION FOR 4-NITRO
CHLOROBENZENE**

(Page 3, clause 0.2) — Insert CAS Registry No. (100-00-5) below the chemical structure.

[Page 4, Table 1, SI No.(iv)] — Insert the following after SI No. (iv):

(1)	(2)	(3)	(4)
v)	Matter insoluble in methanol, percent by mass, <i>Max</i>	0.2	when tested as per clause 11.3 of 'IS 5299 : 2001'

(PCD 11)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR 4-NITRO CHLOROBENZENE

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(Continued on page 2)

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(*Continued from page 1*)

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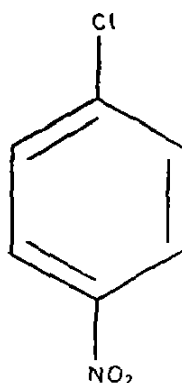
Colour-Chem Ltd, Bombay

Indian Standard
**SPECIFICATION FOR
4-NITRO CHLOROBENZENE**

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 20 August 1976, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 4-Nitro chlorobenzene ($C_6H_4O_2NCl$), also known as *p*-nitro chlorobenzene is an important dye intermediate used in the manufacture of dyestuffs. It is represented by the following structural formula:



4-NITRO CHLOROBENZENE
(Molecular Mass 157.5)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (*revised*).

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 4-nitro chlorobenzene.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of light yellow crystalline mass.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 4-NITRO CHLOROBENZENE

Sl. No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL No. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Crystallizing point, °C, <i>Min</i>	82.4	A-1
ii)	Assay, percent by mass, <i>Min</i>	99.0	A-2
iii)	Dinitro compounds, percent by mass, <i>Max</i>	0.2	A-2
iv)	2-Nitro chlorobenzene, percent by mass, <i>Max</i>	0.5	A-2

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1970*) or as agreed to between the purchaser and the supplier. The containers shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

- Name of the material;
- Name of the manufacturer and his recognized trade-mark, if any;
- Batch number;
- Tare, net mass and gross mass; and
- The minimum cautionary notice worded as under:

‘ BLOOD POISON. AVOID INHALING DUST. DANGER OF CYANOSIS. ’

*Specification for steel drums (galvanized and ungalvanized) (*first revision*).

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE— The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Tests for crystallizing point and assay shall be conducted on each of the individual samples.

4.2.2 Tests for the remaining characteristics, namely, dinitro compounds and 2-nitro chlorobenzene, shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirements of crystallizing point and assay if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of the characteristics tested on the composite sample (see 4.2.2) the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in Appendix A, as indicated in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1960†) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling and tests for dye intermediates.

†Specification for water, distilled quality (revised)

APPENDIX A
(*Table 1 and Clause 5.1*)

METHODS OF TEST FOR 4-NITRO CHLOROBENZENE

A-1. CRYSTALLIZING POINT

A-1.1 Determine the crystallizing point of the material as prescribed in 7 of IS : 5299-1969*.

A-2. ASSAY, DINITRO COMPOUNDS AND 2-NITRO CHLOROBENZENE

A-2.0 Outline of the Method — The contents of 2-, 3- and 4-nitro chlorobenzenes and 2, 4- and 2, 6- dinitro chlorobenzenes are determined by gas-chromatographic analysis.

A-2.1 Apparatus

A-2.1.1 *Gas Chromatograph* — with flame-ionization detector.

A-2.1.2 *Potentiometric Strip-Chart Recorder* — full scale deflection 1 second.

A-2.1.3 *Temperature Programmer*

A-2.1.4 *Column* — made of glass, 2 m long, 6 mm in diameter, and packed with 10 percent Apizon L on 72/85 mesh Celite† or an equivalent material.

A-2.2 Reagents

A-2.2.1 *2, 6-Dinitrotoluene* — internal standard for dinitro chlorobenzenes.

A-2.2.2 *3-Nitrotoluene* — internal standard for mononitro chlorobenzenes.

A-2.2.3 *Chlorobenzene*

A-2.2.4 *2-Nitro chlorobenzene*

A-2.2.5 *3-Nitro chlorobenzene*

A-2.2.6 *4-Nitro chlorobenzene*

A-2.2.7 *2, 4-Dinitro chlorobenzene*

*Methods of sampling and tests for dye intermediates

†These are imported chemicals and are available with the fabricators of gas chromatographs and also with the stockists of chemicals.

A-2.2.8 2, 6-Dinitro chlorobenzene

A-2.2.9 Acetone — solvent.

A-2.3 Procedure

A-2.3.1 *Operating Parameters of Gas Chromatograph*

A-2.3.1.1 *Injection temperature* — 275°C.

A-2.3.1.2 *Column temperature* — 140°C (isothermal) for mononitro chlorobenzenes and 160°C (programmed) for dinitro chlorobenzenes.

A-2.3.1.3 *Carrier gas* — nitrogen, 30 ml/min.

A-2.3.1.4 *Chart speed* — 5 mm/min.

A-2.3.2 *Sample* — Dissolve 10 g of the sample in acetone. Add a known mass of the internal standard and make up the volume to 100 ml with acetone. Inject 0.4 microlitre.

A-2.3.3 *Calibration Factor* — Internal standardization is the method used for calibration of the results of analysis. Make up and chromatograph known mixtures of component 'n' for which the calibration factor is to be determined together with the internal standard plus other components of the sample. The calibration is calculated by the following formula:

$$K_n = \frac{X_n \times A_s}{X_s \times A_n}$$

where

K_n = calibration factor for component 'n' against respective internal standard 's',

X_n = mass percent of component 'n' in the sample,

A_s = peak area for respective internal standard 's',

X_s = mass percent for respective internal standard 's' in the sample, and

A_n = peak area for component 'n'.

A-2.3.4 *Interpretation of Chromatogram* — Elution order of components is as follows:

- a) Acetone (solvent used),
- b) Chlorobenzene,
- c) 3-Nitrotoluene,
- d) Mononitro chlorobenzenes,
- e) 2, 6-Dinitrotoluene, and
- f) Dinitro chlorobenzenes.

Identification is done with the help of test mixtures.

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A-2.4 Calculation

$$\text{Component 'n' in the sample,} \quad \frac{\text{percent by mass}}{= \frac{K_n \times A_n \times X_s}{A_s}}$$

where

K_n = calibration factor for component 'n' (*see* **A-2.3.3**),

A_n = peak area for component 'n',

X_s = mass percent of respective internal standard added to the sample, and

A_s = peak area for respective internal standard.

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